

PATENT APPLICATION
Navy Case No.: 82,502

REMARKS

Claims 18-24, 30, and 31 remain in this application. Claims 25-29 are cancelled by this amendment pursuant to a restriction requirement. Claims 30 and 31 are added by this amendment.

Reconsideration of the application in view of the above amendment and the following arguments is requested.

Claim Rejections – 35 U.S.C. § 102

The Examiner rejected claims 18 and 19 under 35 U.S.C. § 102(b) as anticipated by Duperray et al., U.S. Patent 5,455,225.

Claim 18 is to a composite made by providing a reticulated foam structure comprising a metal selected from the group consisting of silver, silver alloy, gold, and gold alloy; filling the open cells of the foam structure with a high temperature superconducting ceramic oxide or precursor; compacting the filled structure; and heating the compacted structure to melt and/or texture the superconducting ceramic oxide or powder; wherein the metal is selected to have a higher melting temperature than the melt/texture temperature of the superconducting ceramic oxide or precursor.

Duperray discloses a connection between a metal and a solid superconductive ceramic member made by compressing and sintering a body containing superconducting ceramic powder and a silver, gold, or copper foam, partly filled with silver or gold powder and partly filled with superconducting ceramic powder (col. 1, line 66-col. 2, line 13).

Duperray differs from the invention recited in claim 18 in that the superconducting ceramic powder is sintered in Duperray, while the superconducting ceramic oxide or precursor is melted in claim 18.

The attached printout from the McGraw-Hill Encyclopedia of Science & Technology, online at www.accessscience.com, defines "sintering" as "the welding together and growth of contact area between two or more initially distinct particles at temperatures below the melting point." Sintering only causes some localized melting of the surfaces of the particles, but melting as in the present invention causes melting of the entire mass. Thus, by definition, Duperray is not melting the powder as recited in claim 18.

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This difference in the processes results in differences in the products. The structure in Duperray is that of particles fused together. This limits the ability to further process the material into other shapes, as it would tend to fracture along the grain boundaries. This would be detrimental to its connecting function. The present invention is a continuous mass of ceramic oxide and is thus processable.

The present invention is easily able to undergo texturing, which would be much more difficult in Duperray. Texturing can improve the superconducting properties of the material by forming better alignment of the crystals. One type of texturing is to allow the melted superconducting ceramic oxide to undergo anisotropic cooling. For example, heat would be allowed to escape predominantly along one axis. This can create a preferred alignment direction for the crystals, creating better superconductive properties in one direction. However, in Duperray, the portion of the foam that contains ceramic is capped on end by ceramic and on the other end by silver. Duperray would not be able to undergo anisotropic cooling as in the present invention. Further, the crystals in the nonmelted ceramic of Duperray cannot reorient to a preferred direction.

The present invention may also be textured by physical processing into other shapes, such as wires or tapes (p. 8, line 14-p. 9, line 14). This can help to align the crystals in a preferred direction. However, as explained above, processing would not be appropriate for the product of Duperray. Even if such processing were done, the original particles would likely remain intact. This would not result in a preferred crystal alignment.

The present invention further differs from Duperray in that the metal is selected to have a higher melting temperature than the melt/texture temperature of the superconducting ceramic oxide or precursor. New claim 30 specifically recites melting temperature. Duperray does not disclose any relationship between melting points of the ceramic and the metal. All the examples in Duperray use $Y_1Ba_2Cu_3O_7$ (YBCO) and silver foam. The melting point of YBCO is about 1000°C (see attached table from Song, "Experimental Study of a Magnetic Field-Induced Anomalous Microwave Effect in High-Temperature Superconducting Thin Films," Thesis, National University of Singapore, 2001). The melting point of silver is 962°C , which is lower than that of YBCO. The limitation on melting point is not disclosed either expressly or inherently in Duperray.

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The present invention further differs from Duperray in that Duperray's product can only be used as a connector between a solid superconductor and a metal, since they are all made as one body. Any current flowing through it would only flow along the same axis as the metal and the superconductor. There are no means for forming other connections to the connector. However, the present invention can be made in any shape without restriction on how it is used or connected to other components. Current could be flowed through the present invention in any direction.

Claim 30 depends from and contains all the limitations of claim 18 and is asserted to distinguish from the reference in the same manner as claim 18. Claim 19 differs from claim 18 only in that the foam structure is inserted into a sheath in claim 19. The arguments regarding claim 18 also apply to claim 19. Claim 31 depends from and contains all the limitations of claim 19 and is asserted to distinguish from the reference in the same manner as claim 19.

Claim Rejections - 35 U.S.C. § 103

The Examiner rejected claims 20-24 over Duperray in view of Shiga et al., U.S. Patent 5,104,849.

Claim 20 is to a composite made by providing a reticulated foam structure made up of a silver alloy; filling the open cells of the foam structure with a high temperature superconducting ceramic oxide or precursor; compacting the filled structure; and heating the compacted structure to melt and/or texture the superconducting ceramic oxide or powder; wherein the compacted structure is heated to a temperature that is less than the melting temperature of the silver alloy.

Duperray discloses a connection between a metal and a solid superconductive ceramic member made by compressing and sintering a body containing superconducting ceramic powder and a silver, gold, or copper foam, partly filled with silver or gold powder and partly filled with superconducting ceramic powder.

Shiga discloses an oxide superconductor having oxide superconductor layers and metal material layers, which are alternately laminated on each other by a desired number of times (abstract). The metal material layers may be a silver alloy (col. 4, lines 47-53).

In order to make out a *prima facie* case of obviousness under 35 U.S.C. 103, the references must place the invention in the public domain prior to the date of invention, i.e., that the references must generally place the needed subject matter supporting the obviousness

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rejection in the public domain before the date of the invention. *In re Zenitz*, 142 U.S.P.Q. 158, 160 (C.C.P.A. 1964). Even when combined, Duperray and Shiga do not disclose the present invention as recited in claim 20. As explained above, Duperray discloses only sintering, not melting, and can only be used as a connector.

To further make out a *prima facie* case of obviousness, the rejection must be supported by some reason, suggestion, or modification from the prior art as a whole that indicates that the person of ordinary skill would have combined or modified the references. "When the incentive to combine the teachings of the references is not readily apparent, it is the duty of the examiner to explain why combination of the reference teachings is proper ... Absent such reasons or incentive, the teachings of the references are not combinable." *Ex parte Skinner*, 2 U.S.P.Q.2d 1788, 1790 (B.P.A.I. 1987). "It is impermissible to use the claimed invention as an instruction manual or 'template' to piece together the teachings of the prior art so that the claimed invention is rendered obvious." *In re Fritch*, 23 U.S.P.Q.2d 1780, 1784 (Fed. Cir. 1992).

The purpose of using a silver alloy in the present invention is so that the melting temperature of the foam structure can be higher than that of the superconducting ceramic oxide or precursor. This way the superconducting ceramic oxide or precursor can be melted without melting the foam (p. 6, lines 8-13).

Duperray does not melt the ceramic. There is no motivation to use a higher melting metal for the foam, since the foam is not in danger of melting in Duperray. There is no mention of any disadvantage to using pure silver. A person of ordinary skill with knowledge of Duperray would not look to Shiga for a way to raise the melting point of the metal, or to modify the metal in any way.

Shiga recites a list of possible alloys. This list includes some with a higher melting point than silver, such as Ag-Pd, and some with a lower melting point than silver, such as Ag-Mg. Shiga does not disclose a basis for selecting the alloy, or even for using a silver alloy at all instead of pure silver. Shiga does not suggest using a silver alloy with a higher melting point. Further, Shiga is directed to a superconducting material appropriate for cables, while Duperray is directed to a connector for a superconductor. A person of ordinary skill in the art with knowledge of Shiga would not look to Duperray for a method of modifying the cable of Shiga. Neither would he select a high-melting alloy to allow melting of the ceramic.

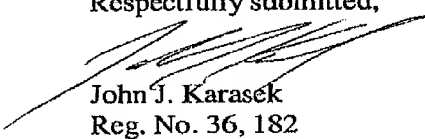
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Claim 20 differs from claim 21 only in that the foam structure is inserted into a sheath in claim 21. The arguments regarding claim 20 also apply to claim 21. Claims 22-24 depend from and contains all the limitations of claim 21 and are asserted to distinguish from the reference in the same manner as claim 21.

In view of the foregoing, it is submitted that this application is now in condition for allowance.

In the event that a fee is required, please charge the fee to Deposit Account No. 50-0281, and in the event that there is a credit due, please credit Deposit Account No. 50-0281.

Respectfully submitted,



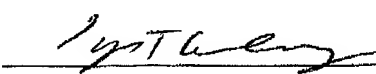
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Sintering

The welding together and growth of contact area between two or more initially distinct particles at temperatures below the melting point, but above one-half of the melting point in kelvins. Since the sintering rate is greater with smaller than with larger particles, the process is most important with powders, as in powder metallurgy and in firing of ceramic oxides.

Powder

Although sintering does occur in loose powders, it is greatly enhanced by compacting the powder, and most commercial sintering is done on compacts. Compacting is generally done at room temperature, and the resulting compact is sub-sequently sintered at elevated temperature without application of pressure. For special applications, the powders may be compacted at elevated temperatures and therefore simultaneously pressed and sintered. This is called hot pressing or sintering under pressure.

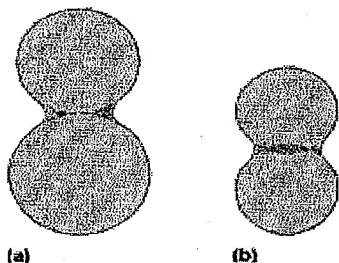
Sintering is observed as an increase in mechanical properties (strength, ductility, and so on) and in many physical properties (for example, electrical and thermal conductivity). In many, but by no means all, sintering processes, the density of the compact increases, that is, the dimensions of the compacts become smaller (the compacts "shrink"), but the shape of the compact is generally preserved. The final density of the sintered compact depends upon the pressure with which the powder is compacted—the higher the pressure, the greater the density—and upon the shrinkage of the compact during sintering. Compacts from a single component powder, for example, a powder of a pure metal or a pure oxide, must be sintered below the melting point of the component, that is, without a liquid phase. Certain compacts from a mixture of different component powders may be sintered under conditions where a limited amount of liquid, generally less than 25 vol%, is formed at the sintering temperature. This is called liquid-phase sintering, important in certain powder-metallurgy and ceramic applications. See also: [Ceramics](#)

Mechanism

The driving force in sintering is surface energy, which decreases because the total surface area decreases as sintering proceeds. When two spheres originally in tangential contact sinter, the area of contact between the spheres increases and a neck is formed. In amorphous materials such as glasses, the process causing neck formation is viscous flow (as when two drops of water coalesce to form one). This type of viscous flow is not possible in crystalline solids. Here the most important material transport process is self-diffusion. Because the material in the neck surface has a highly convex curvature, the number of defects in its crystal structure (vacant lattice sites) is considerably higher than on a flat or concave surface. These defects move by self-diffusion from the convex neck surface to the adjacent flat surface, which means material moves in the opposite direction, that is, the neck grows ([Fig. 1a](#)). Instead of coming from the adjacent flat surface, the material forming the neck may also move by self-diffusion from the grain boundary between the two spheres to the convex neck surface ([Fig. 1b](#)). This latter type of movement explains why compacts shrink, because in this case the centers of the spheres approach each other during sintering. Densification (shrinkage of compacts) is inhibited by rapid grain growth during sintering. During grain growth the grain boundaries, which are the sources of the material which is transported into the convex neck surface, that is, into the pores of the compact, are

swept out of the compact.

Fig. 1 Neck formation. (a) Neck growth through movement of defects from the neck surface to the adjacent flat surface. (b) Movement of neck-forming material from the grain boundary to the neck surface. (After G. H. Gessinger et al., *Continuous observation of the sintering of silver particles in the electron microscope*, *ASM Trans. Quart.*, 61(3):598-604, 1968)



Liquid

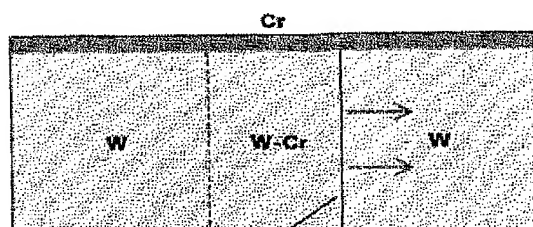
The mechanism of liquid-phase sintering is more complicated. It involves viscous flow of the liquid phase, but also solution of the solid phase in the liquid phase and its reprecipitation in such a way as to make the compact more dense. As in solid-phase sintering, the driving force in liquid-phase sintering is a decrease in surface energy. See also: Cermet; Powder metallurgy

F. V. Lenel

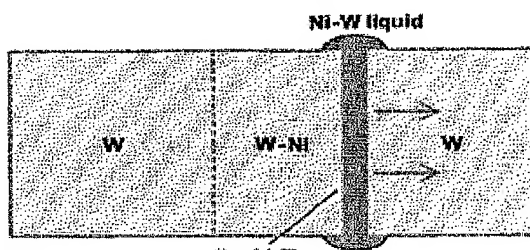
Boundary migration

Powder compacts for sintering are usually made of mixtures of elemental particles, which form alloys as composition change occurs during the sintering treatment. When solute atoms diffuse into the grain boundaries, either present in the original particles or formed in the early stage of the sintering treatment, the grain boundaries may migrate, forming alloyed regions behind them. Such grain boundary migration has been observed in a number of metallic and ceramic materials, and is usually called diffusion-induced grain boundary migration (DIGM) [Fig. 2a]. Solute atoms are supplied from vapor, melt, or solid. During liquid-phase sintering, thin liquid films form between the grains and may migrate if the grains are not chemically in equilibrium with the liquid (Fig. 2b). This phenomenon, called chemically induced liquid-film migration or simply liquid-film migration (LFM), shows characteristics similar to DIGM. Liquid-film migration can be induced either by adding solute atoms to the liquid phase or by changing the temperature. If the amount of liquid in the grain-liquid alloy is large at high temperatures, the solid grains may become corrugated by reprecipitation on certain surface regions and dissolution of adjacent regions. When the composition change by solute addition is large, new grains often form and grow, resulting in diffusion-induced recrystallization (DIR) [Fig. 2c].

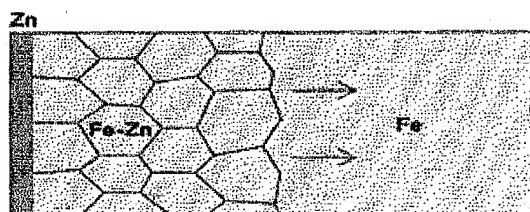
Fig. 2 Boundary migration. (a) Diffusion-induced grain boundary migration in tungsten-chromium (W-Cr). (b) Liquid-film migration in tungsten-nickel (W-Ni). (c) Diffusion-induced recrystallization in iron-zinc (Fe-Zn). The initial positions of the grain boundary and liquid film are shown by broken lines and the migration directions by arrows.



(a) grain boundary



(b) liquid film



(c)

Mechanism

Both DIGM and LFM occur when there is depletion of solute atoms from an alloy, such as by the evaporation of zinc from the surface of a polycrystalline iron-zinc alloy, and both occur at relatively low temperatures, where lattice diffusion of the solute atoms is not rapid. Often the boundaries move against their curvatures. As for any reaction to equilibrium, the driving force for DIGM (and LFM) arises from a decrease in the chemical free energy associated with the composition change. It has been proposed and verified in some experiments that solute atoms diffuse into the grains in front of the moving boundaries and produce coherency strains, which become the driving force for migration. The migration velocity then depends on the size and concentration change of the solute atoms. If the boundaries do not migrate, lattice diffusion is the dominant mode of composition change. The mechanism of forming new grains in DIR is not well understood, but once the grains form, their boundaries move by the same mechanism as for DIGM and LFM.

Applications

Both DIGM and LFM and possibly DIR are found in a variety of practical processes, such as heat treatment, welding, oxidation, sputtering, and melting. In the alloys prepared by liquid-phase sintering, rapid microstructural and compositional changes occur by DIGM and LFM during cooling from the sintering temperature. In electronic ceramics, the boundaries migrate during infiltration with a liquid phase. Such microstructural and composition changes produce significant effects on the resulting properties. The overall composition changes by DIGM, LFM, and DIR in polycrystals, including those produced by sintering, are much more rapid than that expected by diffusion only along stationary boundaries and in the lattice. See also: Alloy; Diffusion

W. Gust

D. Y. Yoon**BIBLIOGRAPHY**

- G. Flight, *Introduction to Ceramics*, 1990
- W. D. Kingery, *Introduction to Ceramics*, 2d ed., 1976
- B. Kubicki, *Sintered Machine Elements*, 1994
- G. C. Kuczynski (ed.), *Sintering Processes*, 1980
- G. C. Kuczynski (ed.), *Sintering*, 1980
- E. Rabkin, C. Y. Ma, and W. Gust, Diffusion-induced grain boundary phenomena in metals and oxide ceramics, in J. Novotny (ed.), *Science of Ceramic Interfaces II*, pp. 353-369, Elsevier, Amsterdam, 1994
- L. H. Yaverbaum (ed.), *Technology of Metal Powders: Recent Developments*, 1980
- D. Y. Yoon, Theories and observations of chemically induced interface migration, *Int. Mater. Rev.*, 40:149-179, 1995

ADDITIONAL READING

- I. Kaur, Y. Mishin, and W. Gust, *Fundamentals of Grain and Interphase Boundary Diffusion*, pp. 343-350, Wiley, 1995
- W. H. Rhee and D. Y. Yoon, *The grain boundary migration induced by diffusional coherency strain in Mo-Ni alloy*, *Acta Metal.*, 37:221-228, 1989

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**EXPERIMENTAL STUDY OF
A MAGNETIC FIELD-INDUCED ANOMALOUS
MICROWAVE EFFECT IN HIGH-TEMPERATURE
SUPERCONDUCTING THIN FILMS**

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(M.Sc., Fudan Univ.; B.Sc., Zhejiang Univ.)

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JAN 06 2003

TECHNOLOGY CENTER 2800

**A THESIS SUBMITTED
FOR THE DEGREE OF DOCTOR OF PHILOSOPHY
DEPARTMENT OF PHYSICS
NATIONAL UNIVERSITY OF SINGAPORE**

2001

Table 2.1 Properties of dielectric substrates used for growth of YBCO films [Moeckly 1994; Lancaster 1997].

Substrate	Structure	Lattice Constant (Å)	% Lattice Mismatch to YBCO	Melting point (°C)	Dielectric Constant @ 300K	Loss Tangent (10^{-6})
LaAlO ₃	Rhomb (perov)	$a=5.36$ $a=3.79$ $c=13.1$	1.6	2110	24	7.6 (77K, 10GHz)
MgO	Cubic (NaCl)	$a=4.212$	9.3	2852	10	6.2 (77K, 10GHz)
Al ₂ O ₃ (α)	Hexagonal (corundum)	$a=4.758$ $c=12.99$	Depend on face	2050	9.3	0.038 (80K, 10GHz)
YBa ₂ Cu ₃ O ₇	Ortho	$a=3.82$ $b=3.89$ $c=11.68$	/	~1000	/	/